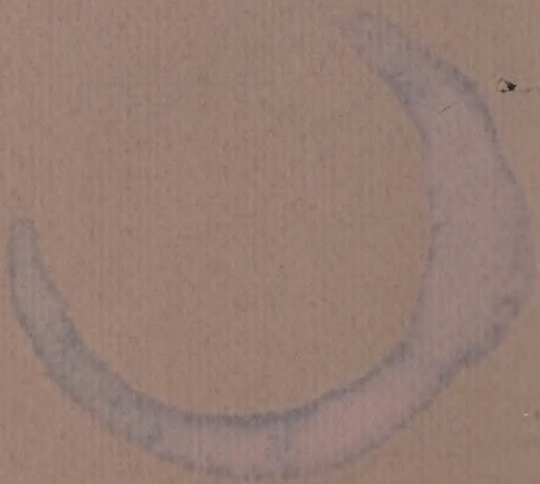
Instruction Manual

Fisher Model 391 K-F Titrimeter®

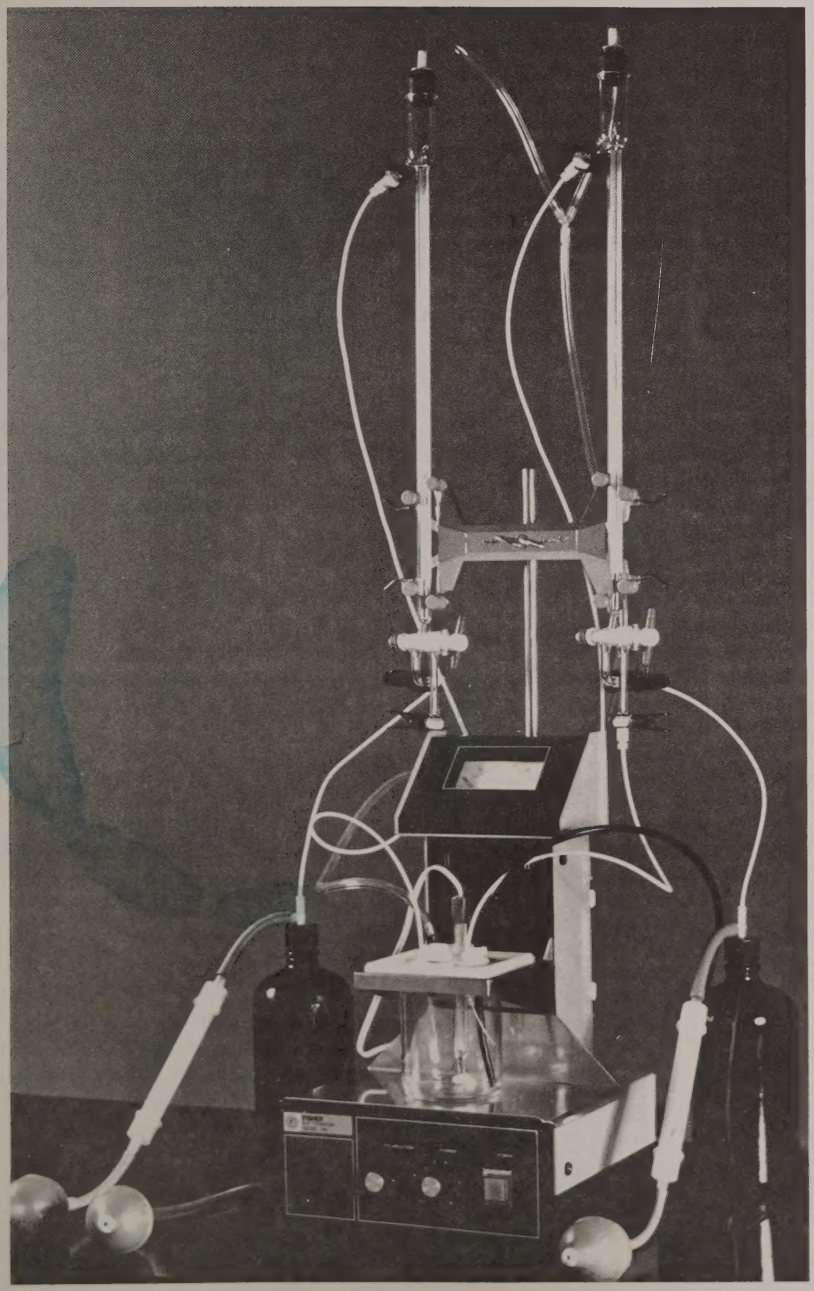
Catalog No. 9-313-130 & -131

MP MASTER
MA33351

INSTRUMENT DIVISION Fisher Scientific Company 711 Forbes Ave. Pittsburgh, Pennsylvania 15219



Fisher
Model 391
K-F Titrimeter®



CONTENTS

PERFORMANCE CHARACTERISTICS	v
SPECIFICATIONS	v
THE INSTRUMENT	1
UNPACKING	3
Packing List	3
Catalog Supplies and Accessories	4
CONTROLS	5
Front Panels	5
Rear Panel	6
ASSEMBLY	7
Major Components	7
Burette Vent Assembly	7
Burette Filling Assembly	9
Final Assembly	10
Optional Second Burette	10
Optional Burette Parts List	11
Optional Burette Assembly	11
OPERATION	13
Filling and Purging the System	13
Setting the POLARIZATION Control	14
Determining the Water Factor of the Karl Fischer Reagent	14
Sample Analysis	15
MAINTENANCE	17
SERVICE	19
Circuit Description	19
Power Supply	19
Meter Circuit	19
Magnetic Stirrer	19
Service Checks	19
Stirring Motor	19
Measuring Circuit	20
Disassembly Procedures	20
Replacement Procedures	21
Replacing Stirring Motor	21
Replacing Microammeter	21
REPLACEMENT PARTS	23

ILLUSTRATIONS

Figure		
1	Base Unit Controls	5
2	Rear Panel Controls	6
3	Assembly of Major Components	8
4	Burette Filling Assembly	9
5	Internal Electrical Components	22
6	Schematic	25

PERFORMANCE CHARACTERISTICS

Accuracy	to 1% of value
Precision	1% rsd
Range	2 to 125 mg H_2O
Sensitivity	20 µg H_2O
Polarizing Current	0 to 50 µA

SPECIFICATIONS

Power Requirements	
Cat. No. 9-313-130	120V, 0.4A, 50/60 Hz
Cat. No. 9-313-131	240V, 0.25A, 50/60 Hz
Dimensions (basic unit, less burettes and tubing)	15 3/4"H x 9 7/8"W x 9 3/4"D (40.01 x 25.08 x 24.77 cm)
Overall Height (with burettes)	50" (127 cm)
Net Weight	~15 lb (6.8 kg)
Shipping Weight	~20 lb (9.1 kg)

THE INSTRUMENT

The Fisher Model 391 K-F Titrimeter® is a bench-top instrument designed to perform the classic Karl Fischer titration for measuring water content in a variety of liquid, gas and solid samples. Basically, the Karl Fischer titration involves a sample with unknown water content and the Karl Fischer Reagent (a mixture of iodine, sulfur dioxide, pyridine and methyl alcohol) as titrant. During the titration, as long as water is present in the sample the iodine in the titrant will be reduced to a relatively colorless (and non-conductive) hydrogen iodide. The moment all the water has been used in this reaction will be the endpoint of the titration, as evidenced by the appearance of free (and highly conductive) iodine from the titrant.

The Model 391 K-F Titrimeter® utilizes an amperometric method of titration to detect this endpoint electrometrically. The method measures current flow in a solution between platinum electrodes connected in circuit with a microammeter and a dc voltage source. The electrodes are placed in the sample solution, a polarizing voltage applied, and the Karl Fischer Reagent slowly added. In the continued presence of water and spent titrant, all current-carrying iodine will have been reduced to iodide and thus there will be little or no indication on the microammeter. As more titrant is added to the sample, the endpoint is eventually reached when no more water is left in the sample to react with the iodine. Any additional titrant simply adds highly conductive free iodine, which causes the microammeter to register the sharply increased current flow--and so indicate the endpoint. Titrant volume used to reach this endpoint is then used to calculate water content in the sample.

The basic K-F Titrimeter® contains a microammeter, integral magnetic stirrer, reaction vessel and support rod, and is supplied complete with burette, electrode, and all necessary tubing and installation hardware. The automatic-zeroing 25 ml burette is held by a clamp to the support rod, while the reaction vessel screws into a special gasketed cover assembly which is hinged to raise for easy installation and change. The cover is fitted with six sealable ports: one small port has a delivery tip to convey titrant into the vessel; a second small port is provided to accommodate a delivery tip should an optional second burette be used; a third port contains a rubber septum for injecting liquid samples; a fourth port secures a waste outlet tube, while the fifth secures an air inlet tube to facilitate evacuation; the sixth port holds the electrode in place. The electrode unit contains dual platinum sensors attached to a single glass body.

Since atmospheric moisture will cause erroneous results in Karl Fischer titration, the system is kept sealed and as dry as possible. The titrant supply bottle has a sealed cap with fittings for an atomizer bulb to pressure-fill the burette. Drying tubes filled with indicating Drierite® are used in all air inlet lines. The top of the burette is fitted with a check valve and plumbed to a drying tube. Tygon® air lines and Teflon® delivery lines complete the air-tight system.

UNPACKING

The K-F Titrimeter® is shipped in a single carton. After unpacking, fill out and return the warranty card, then check each item against the packing list below. If a shortage exists, notify your Fisher branch giving the name and number of the missing article. For necessary reagents, see Catalog Supplies and Accessories below.

NOTE: In the event shipping damage is observed, retain the carton and packing material intact with the instrument and file a claim with the final carrier. Usually, the firm will send an inspector to ascertain liability.

PACKING LIST

Quantity	Item	Number
1	Karl Fischer Titrimeter Assembly	
	120V Unit (Cat. No. 9-313-130)	36443
	240V Unit (Cat. No. 9-313-131)	36853
1	Burette, 25 ml	9-313-132
1	Calibrating Resistor Assembly	39713
1	Rubber Stopper	36444*
1	Rubber Atomizer Assy, Reagent Pump	36531
3	Ball Joint Clamp, Size 12	5-885B
1	Flexaframe® Rod	14-666-10C
3	Drying Tube	9-242B
1	Electrode, Dual Platinum Probe	13-639-123
1	Waste Tube, Polyethylene, 1/4" O.D. x 36"L	36365
1	Coupler, Polyethylene Tubing, 1/4" O.D. x 1 1/2"L	36366
1	Tubing, Vessel to Drying Tube, Tygon,® 5/16" O.D. x 18"L	36361
1	Tubing, Drying Tube to Burette, Tygon,® 5/16" O.D. x 36"L	36362
1	Delivery Tip, Teflon,® Flanged	36378 —
1	Tubing Assy, Burette Overflow, Teflon,® 1/8" O.D.	36377
1	Tubing & Cap Assy, Reagent Supply Bottle**	44293
1	Tubing Assy, Burette to Vessel, Teflon,® 1/8" O.D.	36375
1	Stirring Magnet	9-311-9
3	Coupling, Ball Joint	35238
1	Check Valve	36434
1	Double Burette Holder	5-779
1	Rubber Atomizer	36532
1	Tubing, Atomizer to Drying Tube, Tygon,® 3/8" O.D. x 18"L	36525
1	Tubing, Drying Tube to Reagent Bottle, Tygon,® 3/8" O.D. x 6"L	36526
1	Tray, Stainless Steel	36052
1	Thumbscrew	05915
12	Septum	25908

*Order replacement stoppers under No. 14-135F, Size 4.
**For liter size bottles.

Quantity	Item	Number
1 lb	Indicating Drierite®	7-578-4A
1	Warranty Card	01001
1	Instruction Manual	36411

Catalog Supplies and Accessories

Reagents required for use with the Model 391 are listed below. They are not supplied with the instrument, but should be ordered separately as needed to ensure freshness. Also listed are convenient accessory items, and items that will most commonly need to be replaced.

Item	Number
Karl Fischer Reagent	So-K-3
Karl Fischer Reagent Diluent	So-K-5
Water Standard in Methanol	So-W-2
Burette, 10 ml	9-313-133
Gas Dispersion Tube	9-313-134
Reaction Vessel	9-313-139B
Gasket, Reaction Vessel	9-313-139A
Sample Septum	11-120-1

CONTROLS

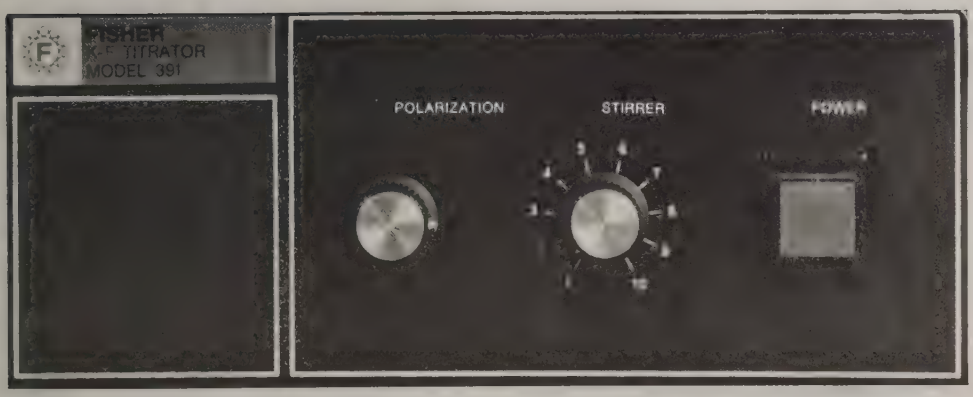
Before assembling and operating the Model 391, refer to figures 1 and 2 and become familiar with the few simple controls.

Front Panels

POWER Switch. This pushbutton switch provides power to the instrument, and is illuminated in the ON position.

STIRRER Control. This rheostat adjusts the rotational speed of the stirring motor. It is calibrated from 1 to 10 in arbitrary units for easy repeatability of settings.

POLARIZING Control. This control sets the level of polarization current (from 0 to 50 μA) as shown on the microammeter.



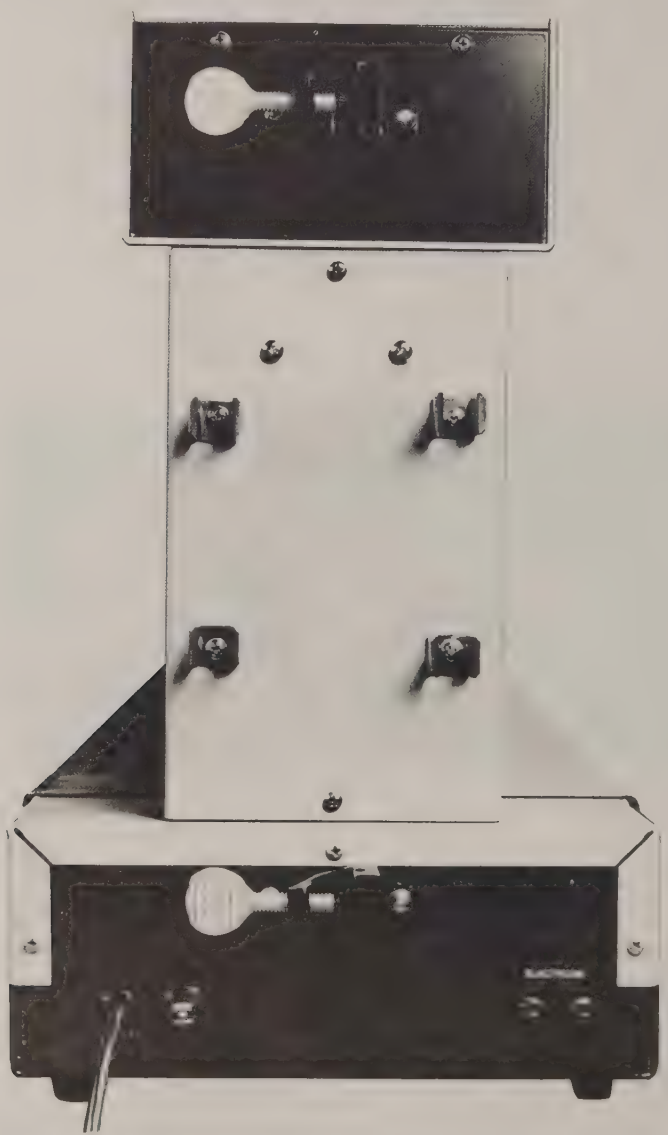
1. Base Unit Controls

MICROAMMETER. This taut-band meter is calibrated from 0 to 50 μA and indicates the current set by the POLARIZATION control. The meter provides the visual indication of the endpoint.

Rear Panel

Electrode Jacks. These jacks receive the plugs from the special dual-sensor platinum electrode.

Fuse. This Slo-Blo fuse is 1/2A in the 120V model and 1/4A in the 240V model.



2. Rear Panel Controls

ASSEMBLY

To ensure proper performance of the Model 391 K-F Titrimeter,® a few assembly procedures must first be performed as detailed below.

Major Components (see figure 3)

1. Secure stainless steel tray to instrument base with supplied thumbscrew.

2. Prepare each of two supplied drying tubes (No. 9-242B) as follows:

   a. Remove both end caps.

   b. Pack small wad of glass wool (Fisher No. 11-388 is recommended) or similar material in one end of tube and replace cap on that end.

   c. Fill tube from opposite end to within one-half inch of top with supplied Indicating Drierite.®

      WARNING: Do not use magnesium perchlorate as a drying agent; an explosive mixture with the reagents used could result.

   d. Pack same material as used in step b above over Drierite,® and replace end cap.

3. Insert prepared drying tubes in spring clips on either side at rear of instrument.

4. Insert Flexaframe® rod (No. 14-666-10C) into two clamps at rear of instrument until about one-half inch of rod extends below lower clamp, then tighten both thumbscrews.

5. Slide double burette holder (No. 5-779) onto upper end of Flexaframe® rod with thumbscrew facing forward, and tighten screw with about one-half inch of rod extending above holder.

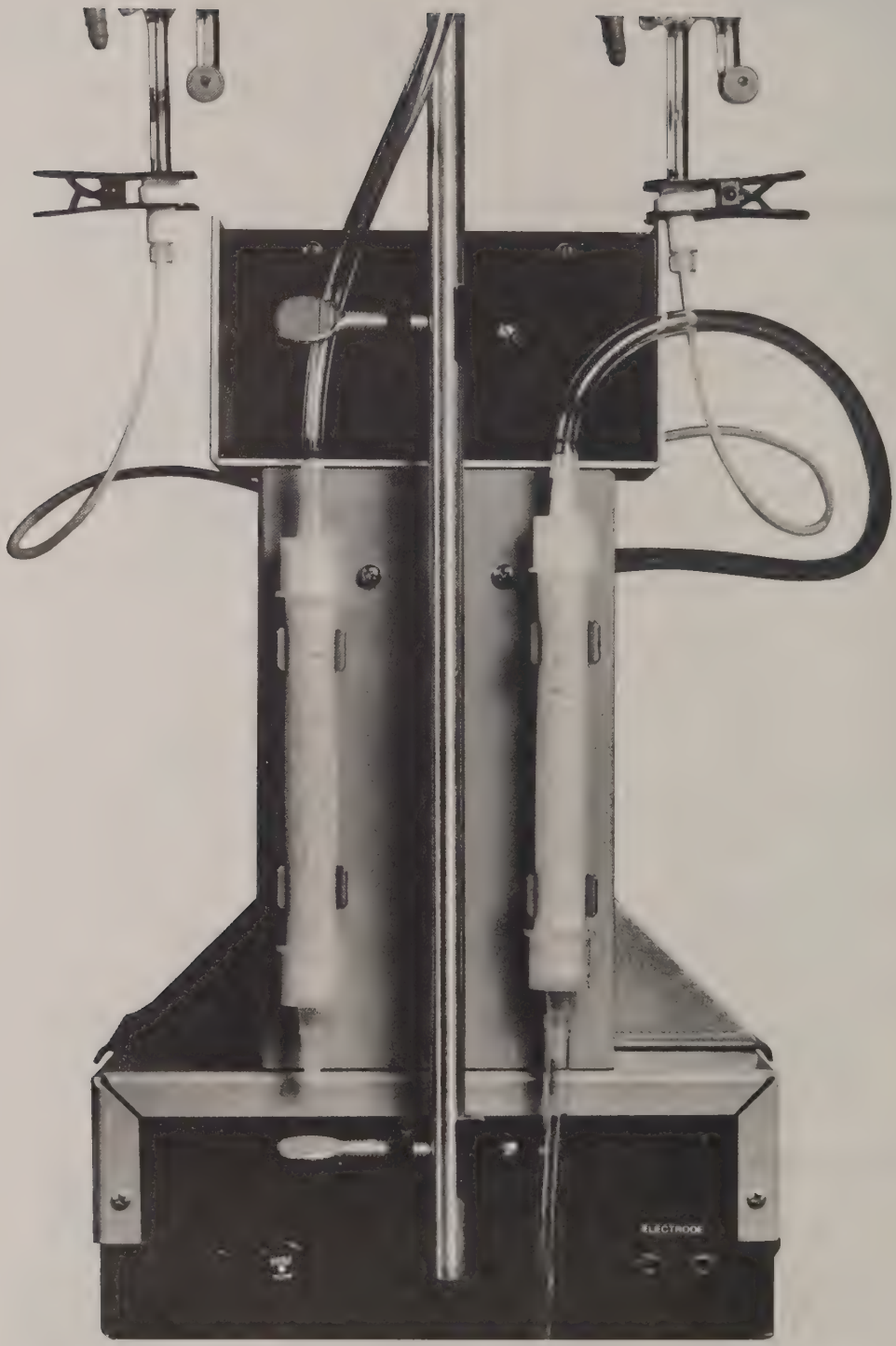
6. Clamp 25 ml burette (No. 9-313-132) in right side of burette holder by compressing scissor-like mechanism, inserting burette, and then releasing mechanism. Position burette with graduations facing forward and the lower tube end about one inch above microammeter housing.

Burette Vent Assembly

7. Insert end of check valve (No. 36434) marked "VAC" into wide end of rubber stopper (No. 36444).

8. To opposite end of check valve attach a 36-inch length of 5/16" O.D. Tygon® tubing (No. 36362).

9. Insert rubber stopper from step 7 in top of burette, and attach tubing from step 8 to top of a drying tube at rear of instrument.



3. Assembly of Major Components

Burette Filling Assembly (see figure 4)

10. Prepare the remaining drying tube (No. 9-242B) as outlined in step 2.

11. To one end of drying tube attach a rubber atomizer assembly (No. 36531).

12. To opposite end of drying tube attach a 6-inch length of 3/8" O.D. Tygon® tubing (No. 36526).

13. Attach tubing from step 12 to the tee of tubing and cap assembly (No. 44293). During operation, this cap will replace the one on the titrant bottle.

14. Screw a ball joint coupling (No. 35238) onto fitting on small diameter tubing of tubing and cap assembly from step 13, then attach coupling to right angle end tube of burette with a ball joint clamp (No. 5-885B).

4. Burette Filling Assembly

Final Assembly

15. Insert 36-inch length of 1/4" O.D. polyethylene tubing (No. 36365) into one of the medium sized ports in the reaction vessel cover assembly to within 1/4 inch of bottom of vessel and tighten gland nut.

16. Insert 1 1/2-inch length of 1/4" O.D. polyethylene tubing (No. 36366) into remaining medium sized port and, leaving about 1/2 inch of tubing extending from port, tighten gland nut. This serves as a coupler.

17. Attach 18-inch length of 5/16" O.D. Tygon® tubing (No. 36361) to coupler from step 16.

18. Attach loose end of tubing from step 17 to top of unused drying tube at rear of instrument.

19. Attach 18-inch length of 3/8" O.D. Tygon® tubing (No. 36525) to bottom of drying tube used in step 18.

20. Attach loose end of tubing from step 19 to rubber atomizer (No. 36532).

21. Insert Teflon® delivery tip (No. 36378) into unused small port in cover assembly.

22. Screw one threaded fitting of Teflon® tubing assembly (No. 36375) into small port containing delivery tip from step 21.

23. Screw a ball joint coupling (No. 35238) onto fitting on opposite end of tubing assembly from step 22, then attach coupling to straight end tube of burette with a ball joint clamp (No. 5-885B).

24. Screw a ball joint coupling (No. 35238) onto fitting of overflow tubing assembly (No. 36377), then attach coupling to overflow outlet of burette with a ball joint clamp (No. 5-885B).

25. Unscrew and remove sample port gland nut (below rubber septum nut in front port of cover assembly) and drop stirring magnet into vessel through opening. Replace and tighten gland nut.

26. Unpackage electrode and slide off clear-plastic protective sleeve from glass body, then carefully insert probe end down through rear port of cover assembly to within 1/2-inch of bottom of vessel (platinum tips must clear stirring magnet) and tighten gland nut.

27. Connect electrode cable to pin jacks at rear of instrument (need to observe polarity) to complete installation.

Optional Second Burette

An optional second burette may be installed into the moisture-tight plumbing system of the Model 391 to dispense any other desired solution (diluted Karl Fischer reagent, Water in Methanol Standard, etc.). To add the second burette, first order the necessary additional parts in the Optional Burette

"

Parts List through your Fisher Branch, and then install the parts as outlined below under Optional Burette Assembly.

Optional Burette Parts List

Quantity	Item	Number
1	Burette, 25 ml*	9-313-132
1	Rubber Stopper	36444
1	Check Valve	36434
2	Tubing, Tygon,® 5/16" O.D. x 12"L	36360
1	Connector, Y	36441
1	Tubing & Cap Assy, Reagent Supply Bottle	44293
1	Rubber Atomizer Assy	36531
3	Ball Joint Clamp	5-885B
3	Ball Joint Coupling	35238
1	Tubing Assy, Burette to Vessel, Teflon,® 1/8"O.D.	36375
1	Drying Tube**	9-242B
1	Tubing, Drying Tube to Reagent Bottle, Tygon,® 3/8" O.D. x 6"L	36526
1	Tubing Assy, Burette Overflow, Teflon,® 1/8" O.D.	36377
1	Delivery Tip	36378

* A 10 ml burette (Fisher No. 9-313-133) may be substituted, if desired.

** Requires Indicating Drierite (Fisher No. 7-578-4A) and glasswool (Fisher No. 11-388). Sufficient material should be left over from assembly of basic instrument.

Optional Burette Assembly

Install the second burette as follows:

NOTE: To simplify the installation instructions, frequent reference is made to the assembly procedures given above for the basic instrument.

1. Install second burette in same manner as detailed in step 6 under Major Components.

2. Insert end of new check valve marked "VAC" into rubber stopper, and insert stopper into top of second burette.

3. Remove 36" length of 5/16" O.D. Tygon® tubing from end of previously installed check valve (other end of 36" tubing should remain attached to drying tube at rear).

4. Connect one end of the two new 12" lengths of Tygon® tubing (No. 36360) to the ends of the check valves.

5. Connect other ends of two 12" long pieces of tubing to arms of Y connector (No. 36441).

6. Connect free end of 36" long tubing (still attached to drying tube at rear) to stem of Y connector.

7. Install second burette filling components by generally following steps 10 through 14 under Burette Filling Assembly.

8. Remove tube end fitting and rubber plug from unused port on reaction vessel cover.

9. Install delivery and overflow components by generally following steps 21 through 24 under Final Assembly.

OPERATION

At this point the operator may refer to any one of a number of ASTM test procedures relative to his sample for full discussion and methodology. A good overview of the Karl Fischer titration method is provided in ASTM E203-64. Note particularly the sections titled "Procedure for Soluble Materials, Either Liquid or Solid," and "Procedure for Insoluble Solids." Also note Appendix III for "Other ASTM Karl Fischer Reagent Water Methods" and Appendix IV for "Determination of Water in Gases."

It is not the purpose of this manual to discuss the above methods, but to provide the general operating requirements involving the Model 391. Specifically, these include: (1) filling and purging the system; (2) setting the POLARIZATION control; (3) determining the water factor of the Karl Fischer reagents; and (4) sample analyses.

Filling and Purging the System

First requirement in operating the Model 391 is to fill the system with reagent and purge it of any trapped atmospheric moisture. Since the operator will be working with the Karl Fischer reagent at this point, a few cautionary words are in order.

CAUTION: (1) Karl Fischer Reagent is composed of four toxic materials. Therefore, avoid inhalation of vapors and contact with skin. Read the safety information on the bottle label.

(2) Karl Fischer Reagent will react with atmospheric moisture to cause a reduction in the reagent's titer. Therefore, do not leave the bottle uncapped any longer than necessary. For the same reason, a filled system should always be securely sealed: tubing and cap assembly on reagent bottle should be checked for tightness, and when instrument is not in use, the burette stopcock should be kept in the closed position.

(3) Any unused Karl Fischer left standing in the burette for more than a few hours will become contaminated. Any contaminated reagent should be discarded and not returned to the supply bottle.

To fill and purge the system, proceed as follows:

1. Remove cap from reagent bottle and immediately replace with tubing and cap assembly.

2. Place burette overflow tube and reaction vessel waste tube into a waste bottle.

3. Turn burette stopcock to fill position.

4. Fill burette with full-strength Karl Fischer Reagent by pumping air into reagent bottle with rubber atomizer. (The small hole in the atomizer tubing must be closed with a finger while pumping the reagent.)

5. Fill burette overflow tube and allow reagent to run into the waste bottle until end of tube is covered. This prevents air from entering the burette through the overflow.

6. Turn burette stopcock to off position and remove finger from hole in atomizer tubing, releasing air pressure from reagent bottle.

   NOTE: If optional second burette is to be used, fill in same manner. (The second burette can hold diluted reagent, or the Water Standard in Methanol if it is used for titer determinations.)

7. Open stopcock on burette containing full-strength Karl Fischer Reagent and allow reaction vessel to fill until platinum electrode tips are covered.

8. Close burette stopcock and empty reaction vessel into waste bottle with rubber atomizer pump.

9. Repeat steps 7 and 8 a couple of times to assure all moisture has been driven from the system.

10. Refill burette from supply bottle as needed.

Setting the POLARIZATION Control

To properly adjust the POLARIZATION control to supply a suitable polarizing voltage, perform the following steps:

1. Remove both electrode leads from the input jacks.

2. Depress POWER switch (pushbutton is illumined in ON position).

3. Insert both pin plugs of the Calibrating Resistor Assembly into the electrode input jacks.

4. Rotate the POLARIZATION knob to cause the panel meter to read approximately 10 microamps.

5. Remove the Calibrating Resistor Assembly and replace the electrode leads in the input jacks.

Determining the Water Factor of the Karl Fischer Reagent

The optimum method for determining the water factor is to titrate known volumes of Water Standard in Methanol (Fisher No. So-W-2). Alternative procedures involve pure distilled water, carefully weighed with syringe addition, or sodium tartarate dihydrate. If the Model 391 is used on a daily basis, the titer need be checked only on a weekly basis. If the instrument is used infrequently, however, normal practice is to check the water factor with each application.

To determine the water factor using Water Standard in Methanol, proceed as follows:

1. Pour 100 to 150 ml of an appropriate solvent into the reaction vessel.

   NOTE: A list of solvents is given under the heading "Reagents" in ASTM E203-64.

2. Adjust STIRRER control for smooth stirring action.

3. Open the burette stopcock and slowly add Karl Fischer Reagent to the solvent until the panel meter indicates that the cell current has reached, and continuously maintained for a period of 30 seconds or more, a reading of 20 μA.

4. Refill the burette.

5. Transfer by pipet 25 ml of Standard Water in Methanol to the reaction vessel through the sample port.

6. Again open the burette stopcock and slowly add Karl Fischer reagent until the panel meter indicates that the cell current has reached, and continuously maintained for a period of 30 seconds or more, a reading of 20 μA.

7. Record the titrant volume dispensed as V, then refill the burette.

8. Compute the water factor F, in milligrams of water per milliliter equivalence, from

$$F = \frac{25}{V}$$

9. Repeat the standardization several times and compute the average water factor.

Sample Analyses

Operating procedures employed for sample analyses are identical to those described above for titrating water standards. Compute results according to

$$\text{ppm } H_2O = \frac{F \bullet V_T \bullet (1000)}{W_s}$$

or

$$\% H_2O = \frac{F \bullet V_T \bullet (0.1)}{W_s}$$

where

V_T = titrant volume dispensed (ml)

F = water factor (mg H_2O/ml)

W_s = sample weight (g)

MAINTENANCE

To ensure the continuance of satisfactory performance from the Model 391 K-F Titrimeter®, the following maintenance procedures should be performed at suitable intervals:

Checking System for Leaks

The plumbing system must be kept airtight; periodically check all lines and connections. In addition, check tubing and cap assembly on reagent bottle or bottles (if two burettes are installed) for tightness, and when instrument is not in use, keep the stopcock or stopcocks in closed position.

Regenerating the Drierite®

The indicating Drierite® should be regenerated in an oven or replaced when the color change (blue to red) indicates near exhaustion of the drying agent.

WARNING: Do not use magnesium perchlorate as a drying agent; an explosive mixture with the reagents used could result.

Eliminating Crystals from System

Crystals may form in the plumbing if the system is left standing with reagent in it for a long period. Care must be exercised to prevent the formation of crystals at connection points and especially in the Teflon stopcocks of the burettes. To remove any crystals that may have formed, a solvent such as acetone, methanol-pyridine (1:1) or methanolchloroform (1:3) can be pumped through the system.

Maintaining the Reaction Vessel

Several components of the reaction vessel will require special attention, as follows:

NOTE: The swing arm bearings on the reaction vessel cover assembly are nylon and do not require lubrication.

1. The O-rings under the gland nuts on the cover assembly should be inspected periodically and replaced when necessary.

2. Keep the magnetic latch clean for positive latching.

3. Because of physical stress and chemical attack, the reaction vessel gasket will gradually deteriorate and will require occasional replacement. To replace gasket, unscrew reaction vessel, remove old gasket from reaction vessel, install new gasket, and screw reaction vessel securely in place again.

   NOTE: Inspect the lip of the reaction vessel for cracks or chips and replace if necessary.

Always handle the electrode carefully to avoid bending the platinum tips. If inadvertently bent, the tips can be straightened using gentle finger pressure; however, exercise extreme care to avoid cracking the glass seal between pins and electrode body.

Even minute cracks can cause an internal short circuit that, in turn, can cause erroneous results.

Moreover, a coating that reduces sensitivity will usually form on the tips after repeated or extended electrode use. This coating can be removed and sensitivity restored by cleaning the tips as follows:

1. Remove electrode from instrument.

2. Immerse electrode to a depth of one inch (but no more than 3/4 of total electrode length) in a solution of Chromic-Sulfuric Acid cleaning solution (Fisher No. So-C-88).

3. Swirl electrode in cleaning solution for about 30 seconds.

4. Rinse electrode with distilled water followed by acetone.

5. Dry electrode thoroughly using a soft tissue.

6. Reinstall electrode in instrument.

SERVICE

The following service information is intended to minimize any downtime and help maintain the instrument in good operating condition. In addition, Fisher service representatives are available to assist with these or any other service problems. Included in this section are complete circuit descriptions, checking procedures, disassembly procedures, and replacement procedures.

Circuit Description

The Model 391 can be ordered for operation from either 120V, 50/60 Hz (Cat. No. 9-313-130) or 240V, 50/60 Hz (Cat. No. 9-313-131). The two units differ by the line cord plug, primary jumpers on the power supply transformer, and the addition of a stepdown autotransformer to the 240 volt version. The schematic diagram (figure 6) shows these differences. The Model 391 includes a polarization current power supply, a simple metering circuit, and a magnetic stirrer.

Power Supply

The polarization current is developed by a printed circuit board power supply. The output of stepdown transformer T1 is rectified by diode CR1 and filtered by capacitor C2. Resistor R1 and diode CR2 develop a regulated 9.5 to 10.5 volts; resistor R2 and diode CR3 produce a further regulated 1.24 to 1.38 volts.

Meter Circuit

Potentiometer R2, a 10-turn unit mounted on the front panel, provides adjustment of the polarization current to the desired level as indicated on microammeter M1. The electrodes, connected across J1 and J2 at the rear of the instrument, sense the resistance of the solution in the reaction vessel. Any change in the resistance of the solution results in a proportional current change at the microammeter.

Magnetic Stirrer

The magnetic stirrer consists of motor MTR1 and speed control rheostat R1. The rheostat provides continuously variable stirring speeds from about 200 to 1650 rpm, depending on the viscosity of the sample solution.

Service Checks

The following check procedures may be used to determine the nature and extent of a malfunction in the stirring motor or the measuring circuit.

Stirring Motor

If the stirring motor still operates, but it is suspected that the stirring speed is not correct, proceed as follows:

1. With a magnetic stirring bar in reaction vessel, place 100 ml of water into vessel.

2. Set STIRRER control to "2" position on dial.

3. Press POWER switch to ON with line cord connected to appropriate power source.

4. Checking with a strobe light, the stirring speed should be near (but less than) 300 rpm.

Measuring Circuit

To check polarization current range and meter circuit, proceed as follows:

1. Disconnect electrode pins at rear of instrument and connect 28.7Ω resistor (1%) to jacks.

2. Set POLARIZATION control to maximum clockwise position.

3. Press POWER switch to ON position with line cord connected to appropriate power source.

4. Microammeter should indicate between 40 and 47 μA.

Disassembly Procedures

To replace electrical components, the following procedures are required for access to base and/or micrometer:

1. Disconnect line cord from power source.

2. Disconnect all tubing connections to burette, then remove burette and set aside.

   NOTE: Before removing any connections, flush Karl Fischer Reagent out of system with clear water.

3. Remove Flexaframe® rod and buret clamp.

4. Gain access to base unit components as follows:

   (a) Remove one screw and plastic washer from either side of base and three screws at rear of base.

   (b) Lift top section from base and set aside.

   NOTE: At this point, all components except microammeter are accessible for replacement or troubleshooting.

5. Gain access to microammeter as follows:

   (a) Remove two screws along top edge at rear of microammeter housing.

   (b) Remove two screws below front edge of microammeter housing.

   (c) Lift out front panel of housing along with microammeter.

Replacement Procedures

All electrical components except the microammeter are mounted inside the base of the instrument as shown in figure 5. Use the Disassembly Procedures to gain access to the component in question. If a circuit board component is involved, remove the four screws that secure the board to the chassis and lift the board out to unsolder the part. Do not unsolder the connecting wires unless the complete board is to be replaced.

Replacing Stirring Motor

1. Perform steps 1 through 4 of Disassembly Procedures.

2. Remove four screws that hold motor to base.

3. Disconnect motor leads and lift motor out of base.

4. Remove magnetic stirring assembly from old motor shaft and attach to replacement motor shaft.

5. Install new motor by generally reversing steps 1 through 3.

Replacing Microammeter

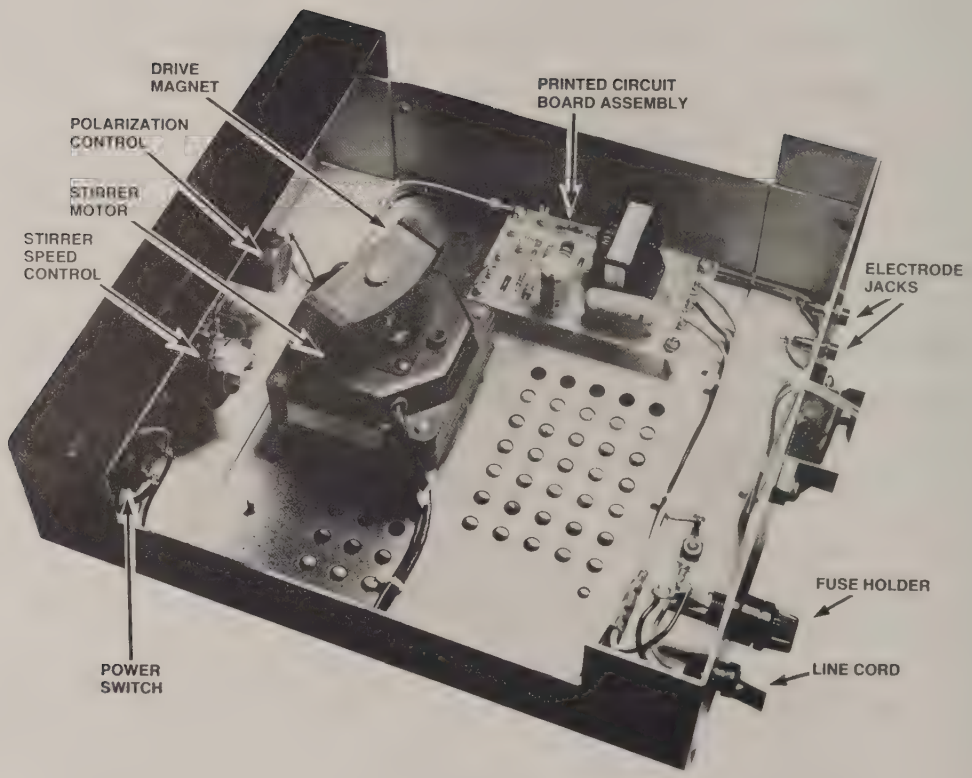
1. Perform steps 1, 2, 3 and 5 of Disassembly Procedures.

2. Carefully remove terminal nuts from rear of microammeter and disconnect leads.

3. Loosen clamping screws at either side of bezel assembly.

4. Lift old microammeter out of bezel and install new meter by generally reversing steps 1 through 3.

   NOTE: Be sure to remove any shorting device used on terminals of new meter.

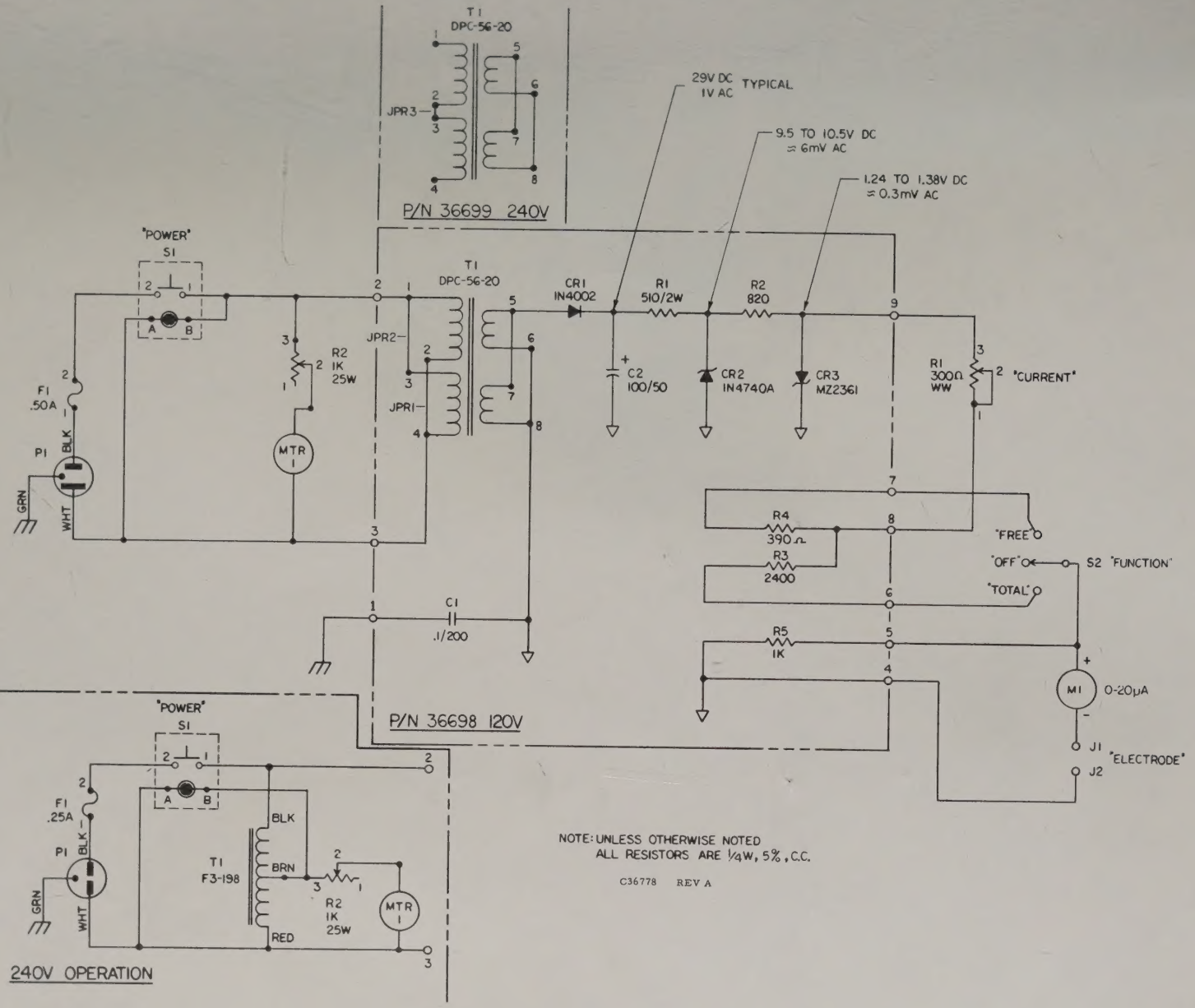


5. Internal Electrical Components

REPLACEMENT PARTS

Item	Part Number	Figure Reference	Schematic Code
Line Cord, 120V	04538	2	P1
Line Cord, 240V	20865	2	P1
Fuse, 120V, 1/2A, Slo-Blo, Buss MDL-1/2	29693	2	F1
Fuse, 240V, 1/4A, Slo-Blo, Buss MDL-1/4	27166	2	F1
Fuse Holder	06296		
Switch, Power	34729	5	S1
Rheostat, Stirrer Speed Control	09259	5	R4
Potentiometer, Polarization Control	26366	5	R3
Knob, Stirrer Speed and Polarization Controls	18953	1	
Data Plate, 120V	36408		
Data Plate, 240V	36868		
Electrode Jacks	20847	3	J1, J2
Rubber Foot	30445		
Tray	36052		
Thumbscrew, Tray Mount	05915		
Clamp, Support Rod	03944	3	
Thumbscrew, Support Rod Clamp	05241	3	
Clip, Drying Tube Mount	06513	3	
Meter	36393		M1
Bezel Kit, Meter	36396		
Motor, Magnetic Stirrer	09275	5	MTR1
Drive Magnet, Stirrer	03936	5	
Autotransformer (240V Model Only)	33807		T1
P.C.Board Assy, 120V	36698	5	
P.C.Board Assy, 240V	36699	5	
NOTE: P.C.Board Assemblies differ only in location of primary jumpers on power supply transformer. Each board consists of the following:			
Capacitor, 0.1 µf, 200V, Sprague 225P10492XD3	36711		C1
Capacitor, 100 µf, 50V, ECI, Type SM	36717		C2
Diode, 1N4002, Fairchild	29876		CR1
Zener Diode, 1N4740A, Fairchild	20759		CR2
Zener Diode, MZ2361, Motorola	36712		CR3
Resistor, 510Ω ±5%, 2W	25680		R1
Resistor, 820Ω ±5%, 1/4W	36710		R2
Resistor, 2400Ω ±5%, 1/4W	36709		R3
Resistor, 390Ω ±5%, 1/4W	42090		R4
Resistor, 1KΩ ±5%, 1/4W	22740		R5
Transformer	36406		T1
Vessel Cover	36358		
Mount, Vessel Cover	36110		

Item	Part Number	Figure Reference	Schematic Code
Nut, Electrode Port	36094		
Nut, Septum Port	36089		
O-Ring, Electrode & Septum Port Nuts	21082		
Septum, Septum Port	25908		
Plug, Septum Port	36298		
Nut, Vent Port	36093		
O-Ring, Vent Port Nut	04447		
Gasket, Vessel Seal	36167		
Tube End Fitting, Optional Burette Port	30961		
Rubber Plug, Optional Burette Port	38335		
Connector Tubing Assy (Tubing & Cap for Reagent Supply Bottle)	44293		
O-Ring, Connector Tubing Assy	21082		



NOTE: UNLESS OTHERWISE NOTED
ALL RESISTORS ARE ¼W, 5%, C.C.

C36778 REV A

Fisher Scientific Co.
Instruments, Apparatus, Furniture and
Chemicals for Laboratories.

Part No. 36411
Operation and Service Procedures
For Model 391 K-F Titrimeter®
Effective with
Serial No. 100 for 120V
Serial No. 100 for 240V
Revised and Published 11-81
Third Issue (1-1202-02)

MP MASTER
MA33351

Litho in U.S.A.